Designation: E544 – 18

Standard Practice for Referencing Suprathreshold Odor Intensity[1]

This standard is issued under the fixed designation E544; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is designed to outline a means for referencing the odor intensities of a material in the suprathreshold region.

1.2 The general objective is to reference the odor intensity rather than other odor properties of a sample.

1.3 This practice is designed to reference the odor intensity on the ASTM Odor Intensity Referencing Scale of any odorous material. This is done by a comparison of the odor intensity of the sample to the odor intensities of a series of concentrations of a reference odorant, for example, 1-butanol (*n*-butanol).

1.4 The method by which the reference odorant vapors are to be presented for evaluation by the assessors is specified. The manner by which the test sample is presented will depend on the nature of the sample, and is not defined herein.

1.5 Test sample presentation should be consistent with good standard practice (**1, 2**)[2] and should be explicitly documented in the test report.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* See Annex A1 for specific safety data.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1292 Test Method for Odor in Water

3. Terminology

3.1 *ASTM odor intensity referencing scale*—series of 1-butanol dilutions used to establish which concentration exhibits an odor intensity matching that of the sample.

3.2 *dynamic scale*—reference scale in which vapor dilutions are prepared by continuous mixing of vapors of 1-butanol with an odorless gas, such as air, to yield constant dilutions of vapor in the gas. An example is presentation through an olfactometer.

3.3 *static scale*—reference scale in which dilutions of 1-butanol in water are prepared in flasks or other suitable containers and presented for odor intensity comparison.

4. Summary of Practice

4.1 The reference odorant used to generate an odor intensity scale is 1-butanol (*n*-butanol). The reasons for its selection are summarized in Appendix X1. A geometric progression scale with a ratio of 2 is recommended, that is, a scale in which each reference dilution differs in its 1-butanol concentration from the preceding dilution by a factor of 2.

4.2 In this method, a series of known concentrations of 1-butanol is used.

4.3 The odor of the sample is matched, ignoring differences in odor quality, against the odor intensity reference scale of 1-butanol by a panel. Assessors report that point in the reference scale which, in their opinion, matches the odor intensity of the unknown.

4.4 The independent judgments of the assessors are averaged geometrically (see 6.3) with respect to the 1-butanol concentrations of the indicated matching points. Results are reported as an odor intensity, in parts per million, of 1-butanol in air (dynamic method) or water (static method) on the ASTM Odor Intensity Referencing Scale. When water is used as a

[1] This practice is under the jurisdiction of ASTM Committee E18 on Sensory Evaluation and are the direct responsibility of Subcommittee E18.04 on Fundamentals of Sensory.

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[2] The boldface numbers in parentheses refer to the list of references at the end of these recommended practices.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

diluent, the temperature of the reference scale solutions during the test must be reported.

4.5 The odor intensity equivalent values which are obtained may then be used to compare the relative intensities of sample groups. These values are reference values and are not related to the odor intensities by a simple proportionality coefficient (see Appendix X2).

5. Procedure

5.1 *Reagents:*

5.1.1 *1-butanol (n-butanol),* the reference odorant, with required purity 99+ mol % by gas chromatography and free of strong odorous impurities.

5.1.2 *Diluent:*

5.1.2.1 *For a "Static Method",* utilizing solutions in a suitable container, 1-butanol is prepared in an aqueous solution using odor-free water.

NOTE 1—If diluent other than water is used, equivalent ppm (vol/vol) values will not exhibit matching odor intensities because of differences in molecular weights, densities, and the activity coefficients of 1-butanol in different solvents. Use of other solvents is therefore not recommended.

5.1.2.2 *For a "Dynamic Method",* utilizing an olfactometer, nonodorous room air, carbon filtered air, or cylinder air are necessary.

5.2 *Preparation of Static Scale:*

5.2.1 Prepare solutions of 1-butanol in water, using pipets and volumetric flasks, following usual laboratory procedures for solution preparation.

5.2.2 *Procedure*—Place the reference sniffing solutions into a suitable glass flask or jar. The volume of solution should be selected to not fill more than ¼ of the sniffing container headspace. Between sniffing, cover the top of each container in order to assure equilibration between the solution and the air head-space above it. The container should be gently swirled by each assessor prior to each sniffing in order to assure equilibrium.

5.2.3 The temperature of the reference solutions during the test should be ambient and kept constant during the test.

5.2.4 The odor threshold of 1-butanol in water is 2.5 ppm at 21°C (**3**). The useful concentration range for the static scale is above this value but does not extend to the solubility limit of 7.08 % of 30°C (70 800 ppm) (**4**). At concentrations close to the solubility limit, excess 1-butanol may separate from the solution with temperature change. If this occurs the odor becomes equivalent to that of pure 1-butanol.

5.2.5 Considerable latitude as to the selection of concentrations is allowed. To go from the saturation point to the threshold requires 16 steps, assuming that each succeeding mix is one half of the preceding concentration (70 800; 35 400; 17 000; 8850 ppm; etc.). Solutions stronger than 20 000 ppm of 1-butanol exhibit an odor that is too intense for most comparisons.

5.2.6 The most useful concentration range is approximately between 10 and 20 000 ppm, and may be covered by twelve flasks containing 10 ppm in flask No. 1, 20 ppm in flask No. 2, etc. These flasks constitute the static scale.

5.2.7 The scale points are arranged systematically, in the order of increasing concentrations, and are numbered in ascending integers, from 1 for the lowest concentration of 1-butanol.

5.3 *Preparation of Dynamic Scale:*

5.3.1 Prepare the 1-butanol airflow mixtures in an olfactometer apparatus utilizing a known concentration of butanol from a prepared gas cylinder or by a headspace over liquid butanol.

5.3.2 An adequate concentration range will depend on olfactometer design, for example, presentation air flow rate. For most applications a range between 200 ppb and 2000 ppm of 1-butanol in air is most useful.

5.4 *Reference Concentrations:*

5.4.1 This practice is intended to establish, on a continuous 1-butanol scale, that concentration which best corresponds in its odor intensity to the odor intensity of the sample. Since a continuous scale is technically difficult, a common olfactometer design consists of a series of discrete concentration points at sniffing ports delivering known concentrations of 1-butanol vapor in air.

5.4.2 A geometric progression scale of concentrations is used, in which each reference port differs in its 1-butanol concentration from the preceding port by a factor of 2.

5.4.3 The scale points are arranged systematically, in the order of increasing concentrations, and are numbered in ascending integers, from 1 for the lowest concentration of 1-butanol.

5.5 *Test Room*—The test room must be well-ventilated, essentially odor-free, and comfortable. In order to avoid bias, waiting assessors should not observe or learn the judgments of the assessor currently matching the odor intensity of the sample to the scale (**1, 2, 5, 6**).

5.6 *Odor Panel:*

5.6.1 *Number*—The number of assessors should be sufficient to permit proper statistical tests on their judgments. A minimum panel of six assessors is recommended. A small number of assessors may be used with replicate judgments to increase the total number of responses. It is important when obtaining replicate data that all bias is removed. Precautions such as separate sessions and recoding are recommended (**1**).

5.6.2 *Selection*—Extensive training is not needed but precautions must be taken in the selection of the assessors (**7, 8**). An individual with insufficient sensitivity to detect the odor of 10 ppm of 1-butanol in water should not be an assessor. Also, some individuals have been observed to experience difficulty in matching odor intensities. Prospective assessors can be screened by having them repeatedly match the odor intensity of a known concentration of 1-butanol to the 1-butanol reference scale. Those whose standard deviation in repeated testing exceeds 1.5 scale steps should not be used in the panel. Periodic retesting of assessors is advisable.

5.7 *Judgment Procedure:*

5.7.1 Assessors are instructed on the nature of the 1-butanol odor intensity reference scale. They are told that the reference points are numbered beginning with No. 1, which represents

the weakest odor and that the odors increase systematically in intensity with increasing identification numbers.

5.7.2 Assessors are instructed to smell the unknown sample and then to smell one or two references on the scale as they feel necessary. They should begin with weakest references, and match the unknown to the scale, ignoring differences in the odor quality. They are permitted to check and recheck the unknown against the scale any number of times and should not be hurried or biased by others in any manner.

5.7.3 Assessors should be instructed to take small, controlled sniffs of the test samples and the references. It is important for assessors to be consistent in their sniffing technique when evaluating the samples and also sniffing the references for comparison.

5.7.4 Assessors are advised that they may report one of the scale points as the best match, or they may report that the best match occurs between two adjacent points, for example, the unknown is stronger than scale point No. 7, but weaker than scale point No. 8.

5.7.5 Assessors should be advised that the odor may also be weaker than the weakest point of the scale, or stronger than the strongest point of the scale.

5.7.6 When the judgment is within scale limits, the assessor should make sure that the selected position is a good match, that is, that the next lower concentration of 1-butanol indeed smells weaker than the unknown, and that the next higher concentration indeed smells stronger.

5.7.7 Assessors report the matching point in terms of the port identification number. When the best match is a position between the scale points, such as between port Nos. 7 and 8, the half-number, 7.5, may be used.

5.7.8 During repeated smelling of one or more samples or 1-butanol scale points, olfactory adaptation can occur, rendering the sense of smell less sensitive (**9**).

5.7.9 Because of this olfactory adaptation, an assessor may find that after judging at higher odor intensity points on the scale, they may have difficulty in detecting odor at the lowest points of the scale. A rest of 2 to 5 min will usually correct this effect.

5.7.10 Assessors may differ in the amount of time required to render a judgment. The assessor should be allowed to proceed at a comfortable rate. As many as six test stimuli can be handled by a panel of up to ten in a 1-hour session.

6. Calculation

6.1 The values of parts-per-million concentrations of 1-butanol in water solutions or air are known from the method of preparation (see 5.2 and 5.3).

6.2 When an assessor indicates that a position between two scale points is the best match, the concentration value for this position is calculated as the geometric mean of the concentrations at the two adjoining scale points. This applies to both procedures. For example, if the 1-butanol concentrations at points No. 7 and No. 8 are 658 and 1280 ppm, then the concentration that would correspond to the intermediate position of 7.5 is found by the following logarithmic computation:

$$\log(\text{ppm for position } 7.5) = \frac{\log(658) + \log(1280)}{2} = 2.96 \quad (1)$$

The antilogarithm gives 918 ppm as the estimate for the 1-butanol concentration at the scale position 7.5.

6.3 *Averaging Assessors' Data*—A geometric average of a group of assessors' judgments is computed and converted into an ASTM Odor Intensity Referencing Scale value, in parts per million of 1-butanol in water, in a manner illustrated by the following example:

6.3.1 The odor of a test sample prepared in 125-mL Erlenmeyer flasks was evaluated for its odor intensity in comparison to that of a prepared static scale. Nine assessors participated.

Assessor	Matching Sniffing Port No.	1-Butanol Concentration Data ppm (vol/vol)	log (ppm)
1	5	165	2.22
2	6.5	452	2.66
3	7	658	2.88
4	6.5	452	2.66
5	7.5	919	2.96
6	7.5	919	2.96
7	7.5	919	2.96
8	6.5	452	2.66
9	5.5	226	2.35

6.3.2 The mean $\log_{10}$ in parts per million was equal to 2.694. The antilogarithm of 2.694 is 494 ppm of 1-butanol. This would be the best mean for the odor intensity match for the test sample. This result should be reported in accordance with Section 7.

6.4 *Standard Deviation*—It is desirable to provide the standard deviation of the mean $\log_{10}$ (ppm) value (**6**), for the method of calculation used when reporting the results. For the example given in 6.3.1, the standard deviation of the mean log (ppm) of 2.694 is ±0.27.

7. Report

7.1 When the diluent is water and the static-scale method is used, report the result as follows:

7.1.1 The odor intensity of the sample is equivalent to __ ppm of 1-butanol in water, __ °C, in the ASTM Odor Intensity Referencing Scale.

7.2 When a dynamic scale method is used, report the results as follows:

7.2.1 The odor intensity of the sample is equivalent to ___ ppm of 1-butanol (air) on the ASTM Odor Intensity Referencing Scale.

7.3 Report the standard deviation of the result (see 6.4), if it is calculated. Also report the number of assessors that participated and if evaluations were replicated.

7.4 Values that are reported in this manner permit the comparison of odor intensity measurements for the same material to be conducted in different locations by different panels, the comparison of odor intensities for samples which are not available at the same time, and the reconstruction of a reported odor intensity for an unknown material in other laboratories.

8. Keywords

8.1 *n*-butanol; supra threshold odor intensity